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Polymer light-emitting diodes with doped hole-transport layers

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Dedicated to the memory of Professor Bert de Boer, who passed away in January 2009.

Keywords hole-transport layer, light-emitting diodes, polymers, poly(phenylenevinylenes)

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We demonstrate a solution processed bi-layer PLED based on poly(p-phenylene vinylene) derivatives using orthogonal solvents. To lower the voltage drop the hole transport layer (HTL) based on poly[2,5-bis(2′-ethylhexyloxy)-*co*-2,5-bis(butoxy)-1,4-phenylenevinylene] (BEH/BB-PPV (1:3)) is doped with tetracyano-tetrafluoro-quinodimethane (F4TCNQ). The conductivity of BEH/BB-PPV (1:3) was observed to increase by two orders of magnitude upon doping with F4TCNQ. The doped HTL was observed to lower the operating voltage of a double layer PLED, but suffers from additional quenching by the dopant at higher voltages due to the lack of an electron blocking functionality.

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1 Introduction Polymer light-emitting diodes (PLEDs) are attractive due to their potential for cheap fabrication via roll-to-roll processing. Currently, most PLEDs consist of only one active polymer layer that governs all processes necessary for light generation in the device: charge injection, transport and recombination. Due to the low charge carrier mobility of typical light-emitting polymers, devices have to comprise of relatively small layer thicknesses, typically around 100 nm, in order to keep the operating voltage low. For roll-to-roll coating techniques such as inkjet printing and slot-die coating it is very difficult to operate in such a small process window and uniform layer thicknesses of ∼100 nm without defects are difficult to achieve with these techniques [1]. Inevitably, these processing techniques are very susceptible to creating defects on large-area devices, leading to short circuits between the electrodes. Furthermore, in most of the commonly used electroluminescent polymers the electron and hole transport are highly unbalanced. In conjugated polymers such as poly(2-methoxy-5-(2′-ethyl-hexyloxy)-p-phenylene vinylene) (MEH-PPV) the electron current is limited by trapping and hence orders of magnitude lower than the hole current, which can be regarded as trap-free. As a result of the highly unbalanced charge transport, excitons are mainly formed near the electron-injecting cathode. It has been shown that within ∼10 nm of the cathode exciton quenching occurs, which can be regarded as a non-radiative recombination process [2]. Therefore, exciton quenching is a major loss mechanism in thin layer PLEDs.

An attractive way to overcome these issues is the fabrication of multilayer PLEDs, in which separate layers are responsible for injection and recombination. Using separate injection layers for holes and electrons, the recombination can be confined to the emissive layer, reducing quenching from the metal electrodes. By employing this method, the efficiency and life time of the devices can be significantly improved. Furthermore, a thicker multilayer structure makes the devices more robust with regard to electrical shorts. Currently, state-of-the-art multilayer light-emitting diodes based on small organic molecules are usually fabricated by thermal evaporation of the layer sequence in a n–i–p architecture [3–5]. Power efficiencies of 90 lm/W have

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already been achieved for a white-emitting stack [6]. Alternatively, solution processing is a more cost-efficient alternative compared to thermal evaporation techniques for which a high vacuum is required. However, wet-processing of multilayer devices is troublesome since the solvent used for processing the consecutive layer may damage or even dissolve the existing layers.

Up to now, various approaches have been proposed to fabricate multilayer PLEDs. Water or alcohol-soluble polyfluorene derivatives or highly solvent specific polyurethane have been synthesized [7–11]. These materials can be spin casted on top of the emission layer (EML) as the electron transport layer (ETL) because of their solvent compatibility. Multilayer structures can also be achieved by blade coating [12, 13], dip-coating [14] or crosslinking [15]. Recently, Tanase et al. [16] presented a double layer PLED using an orthogonal solvent system. The tailor-made copolymer poly[2,5-bis(2′-ethylhexyloxy)-*co*-2,5-bis(butoxy)-1,4-phenylenevinylene] (BEH/BB-PPV (1:3)) (Fig. 1) was designed to function as the bottom hole transport layer (HTL). The very short bis(butoxy) side-chains on the BB-component reduce the solubility of the copolymer rendering it soluble in chloroform and insoluble in toluene. Furthermore, the symmetric side chains in BEH/BB-PPV (1:3) reduce the structural disorder of this material, and thereby enhance the charge transport. However, in spite of the enhanced hole mobility of BEH/BB-PPV (1:3) (10^{-9} m^2/Vs) it turned out that 1/3 of the applied voltage potential was still lost in the HTL. To overcome this problem it is necessary to increase the conductivity of the HTL by p-type doping. Several dopants have been observed to cause p-type doping in organic materials, such as the transition metal oxides MoO_3 [17], ReO_3 [18] and WO_3 [19]. However, these inorganic molecules are generally incompatible with solution processing from organic solvents. Moreover, the small size of these molecules makes them susceptible to migration across the organic layers, where the negatively charged dopants may cause exciton quenching in the EML. Large organic compounds, such as BAPD [20] and F2-HCNQ [21], are likely to be less mobile in the polymer matrix. Among them, tetracyano-tetrafluoro-quinodimethane (F4TCNQ) has proven to be a very effective p-type dopant for organic small molecules [22, 23] as well as solution processed polymers [24, 25]. It has been reported that the hole transport can be increased by several orders of magnitude upon doping. In this report, we demonstrate that by solution-based p-doping of the HTL, the conductivity of this layer can be greatly improved, leading to a lower operating voltage of a bilayer PLED.

2 Experimental MEH-PPV and BEH/BB-PPV (1:3) were synthesized and purified as described previously [16]. F4TCNQ was purchased from Sigma-Aldrich and used without further purification. MEH-PPV, BEH/BB-PPV (1:3) and F4TCNQ were dissolved in toluene, chloroform and NMP, respectively. The BEH/BB-PPV (1:3) and F4TCNQ blend solution were made in a nitrogen atmosphere to avoid contamination with water and oxygen and kept at room temperature until the aggregation completely disappeared (around 1 h). The spin-coated thin films were uniform and transparent and the light generated by the completed device was homogeneous.

Glass substrates prepatterned with ITO were cleaned in an ultrasonic bath with acetone and iso-propanol. After a UV-ozone treatment for 20 min, the ITO substrates were covered by a ~55 nm PEDOT:PSS (H.C. Starck) layer and baked at 140 °C for 15 min. The polymer layers were subsequently spin-coated under nitrogen atmosphere. For the hole-only devices, 20 nm Pd and 80 nm Au layers were deposited as top contacts by thermal evaporation under vacuum (1×10^{-6} mbar). For the PLEDs, 5 nm Ba and 100 nm Al layers were deposited at 2×10^{-7} mbar. The polymer thicknesses were measured using a Dektak 6 M profile analyser. The current–voltage characteristics of the devices were measured using a Keithley 2400 source meter. The light output was recorded simultaneously using a calibrated Si photodiode connected to a Keithley 6514 electrometer.

3 Result and discussion Due to its selective solubility, BEH/BB-PPV (1:3) is a particularly good candidate for the HTL in multilayer PLEDs and therefore used as host material in this study. In order to increase its transport properties F4TCNQ was selected as the dopant molecule. A major complication in the fabrication of a solution processed p-doped film is that the charge transfer between host and dopant already takes place in solution. As a result, the polymer host strongly aggregates or even precipitates. In both cases the solution cannot be processed anymore into functional thin films. Therefore, realization of a spin casted polymer film with a controllable amount of doping is a considerable challenge that will be addressed first.

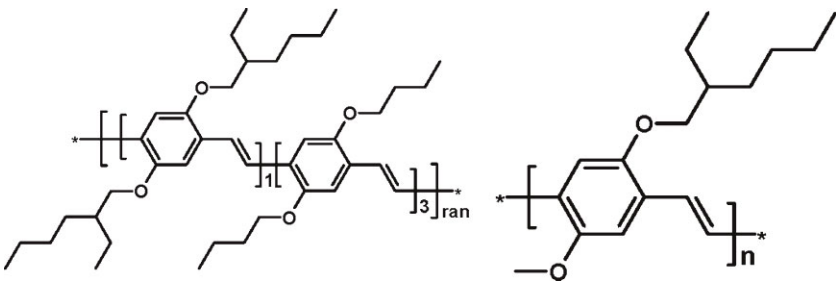

Figure 1 Chemical structures of the polymers: BEH/BB-PPV (1:3) and MEH-PPV.

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As a first step, the polymer and the dopant were dissolved in different solvents and mixed subsequently. To suppress the aggregation in the composite solution, it is of importance that several issues are considered, such as the molecular weight of the polymer, the concentration of the polymer in solution, the doping concentration, the solvent for the dopant, the polarity and the viscosity of the solvents and the temperature of the composite solution. The hole transporting polymer BEH/BB-PPV used in this study is highly solvent specific, requiring the use of chloroform as solvent. Since the polarity of chloroform is much higher than that of other commonly used organic solvents, such as toluene, the previously published recipe by Zhang et al. [25] is not applicable anymore. The dopant F4TCNQ requires a polar solvent, in this study dimethylformamide (DMF), N-methyl-2-pyrrolidone (NMP) and tetrahydrofuran (THF) were tested. Figure 2 shows the absorption spectra of the three F4TCNQ solutions. The peak centred at ~400 nm appears in all the three spectra, which corresponds to the main absorption band of a single F4TCNQ molecule. However, the peak around 800 nm appears only in the F4TCNQ in DMF and THF spectra. For the F4TCNQ in NMP solution, the low energy features are completely absent in the absorption spectrum. The absorption at 800 nm most likely originates from the F4TCNQ radical anion, which is generated from the redox reaction with water present in THF and DMF [26]. The formation of the F4TCNQ radical anion reduces the effectiveness of F4TCNQ solution for doping. Considering this feature, we used NMP as the solvent for F4TCNQ.

As a next step, the F4TCNQ/NMP solution was added to the BEH/BB-PPV/chloroform solution. It was found that by using NMP as solvent for the dopant the precipitation in the polymer/dopant composite solution can be largely suppressed. The composite solution was kept at room temperature until the aggregation fully disappeared. Subsequently, the solution was spin casted on glass substrates without further filtering, yielding transparent and uniform polymer

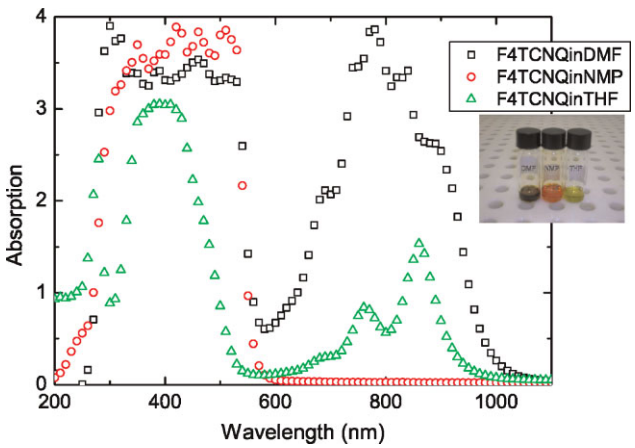

Figure 2 (online colour at: www.pss-a.com) The absorption spectra of F4TCNQ in three different solvents: DMF, NMP and THF. All the solutions have the same concentration: 0.5 mg/ml.

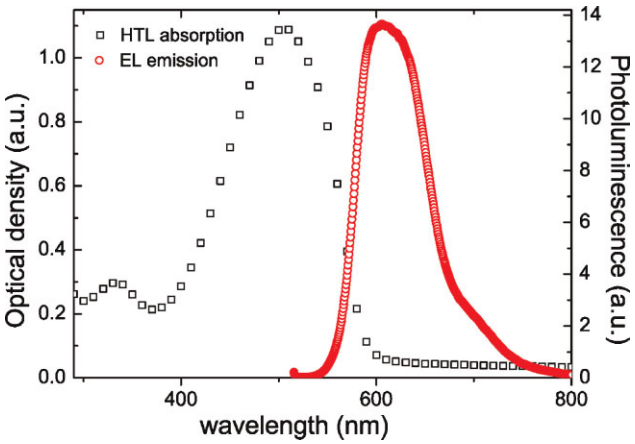

Figure 3 (online colour at: www.pss-a.com) Normalized optical density (squares) of BEH/BB-PPV (1:3) and PL spectra (circles) of MEH-PPV thin films.

layers, of which the absorption spectra were measured. As evidenced by photoluminescence (PL) and absorption spectra, the overlap between the absorption of F4TCNQ doped BEH/BB-PPV (1:3) and the emission of an MEH-PPV thin film is small (Fig. 3), suggesting that absorption by the HTL in a double layer device is negligible when MEH-PPV is used as the EML. In order to investigate the transport properties of the doped HTL, hole-only devices were fabricated using a Pd/Au cathode to block the injection of electrons. The anode consists of indium tin oxide (ITO) covered with a spin coated layer of poly(3,4-ethylenedioxythiophene):poly(4-styrene sulphonate) (PEDOT:PSS). The hole current could be enhanced by one order of magnitude using a doping ratio of (1:100), indicating that F4TCNQ acts as a p-type dopant for BEH/BB-PPV (1:3) (Fig. 4). Under reverse bias the current density of the undoped device is lower than under forward bias, due to the injection barrier

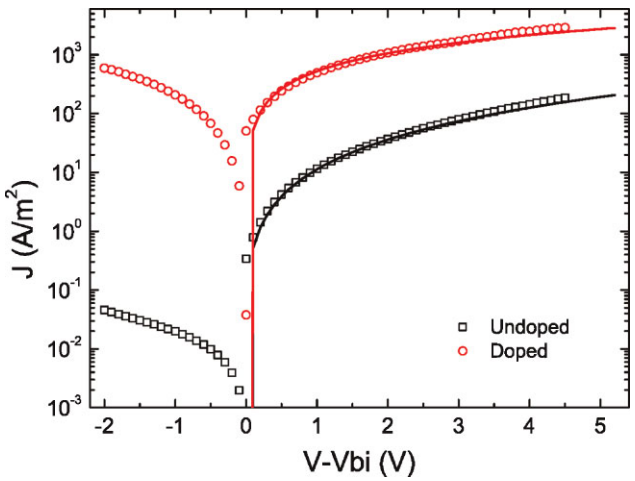

Figure 4 (online colour at: www.pss-a.com) *J–V* characteristics of undoped and doped BEH/BB-PPV (1:3) hole only devices. All the devices have the same thickness: 250 nm. The solid lines represent the prediction of the device model.

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between Pd (−4.8 eV) and the highest occupied molecular orbital (HOMO) of BEH/BB-PPV (1:3) (−5.35 eV). In contrast, the current density–voltage (*J*–*V*) characteristics of a doped device are symmetric. The strong increase of the hole current under reverse bias stems from a dipole at the organic/metal interface induced by the adsorption of F4TCNQ on Pd, which in turn reduces the injection barrier [27, 28]. To characterize the charge transport first the undoped devices were modelled using a numerical drift-diffusion device model which incorporates a charge carrier density dependent mobility [29, 30]. A hole mobility of 3×10^{-9} m^2/Vs for the undoped BEH/BB-PPV is obtained at low fields. To describe the characteristics of the doped devices, one parameter is added to the model, namely p_0, which is the density of the free charge carriers generated through doping. From the modelling we obtained a value of p_0 of 2×10^{23}/m^{-3}.

As a next step double layer PLEDs were fabricated with a 40 nm F4TCNQ doped BEH/BB-PPV (1:3) layer as the HTL and a 60 nm MEH-PPV layer as the EML. Two different control devices were fabricated in the same batch for comparison: single layer devices comprising only the EML and double layer devices with an undoped HTL. Figure 5 displays the *J*–*V* characteristics of the three device structures. The current, the luminance (*L*) and the conversion efficiency (light output/current) of the single layer 60 nm device are higher than that of the double layer devices. The increase in current, and hence luminance, can be understood from the small thickness of the device, causing an enhanced electric field across the layer. In this regard, a significant difference would be expected between the current of the double layer devices with a doped and undoped HTL, which, however, is not the case. In order to understand this we modelled the double layer PLEDs with and without doping (the solid lines in Fig. 5a and b). For the electron transport an exponential distribution of electron traps was taken [31]. The calculated 100 nm single layer device characteristics (the dashed lines in Fig. 5a and b) are also presented as a reference. The charge carrier mobility of MEH-PPV and BEH/BB-PPV (1/3) were determined from modelling of the single carrier devices. By combining both layers and using their respective parameters, the characteristics of the double layer device without doping can be calculated. For the PLED with doped HTL we used the value of p_0 that was deduced from the *J*–*V* characteristics of a p-type doped HO device, as shown in Fig. 4. With the numerical description of the *J*–*V* characteristics also the electric field distribution inside the double layer devices can be calculated. Figure 6 shows the electric field at 2.5 V. At $x = 0$ and $x = 100$ nm the polymer layer contacts with the cathode and the anode, respectively. The solid and the dash curves are the calculations with $p_0 = 2 \times 10^{23}$ m^{-3} (doped HTL) and $p_0 = 0$ (undoped HTL), respectively. When the doping concentration p_0 is zero, the electric field across the HTL is nonzero, indicating that there

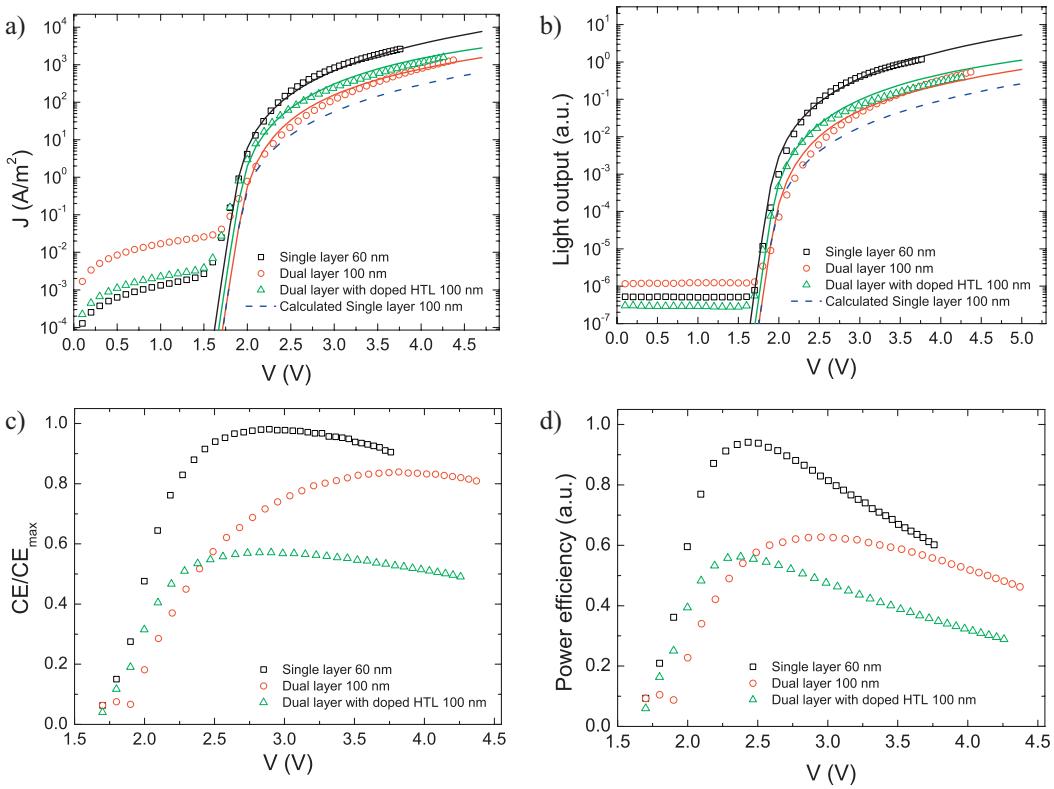

Figure 5 (online colour at: www.pss-a.com) (a) *J*–*V*, (b) *L*–*V*, (c) normalized conversion efficiency (light output/current) and (d) normalized power efficiency (light output/($J \times V$)) characteristics of single layer and double layer PLEDs. The solid and dash lines represent the numerical simulation.

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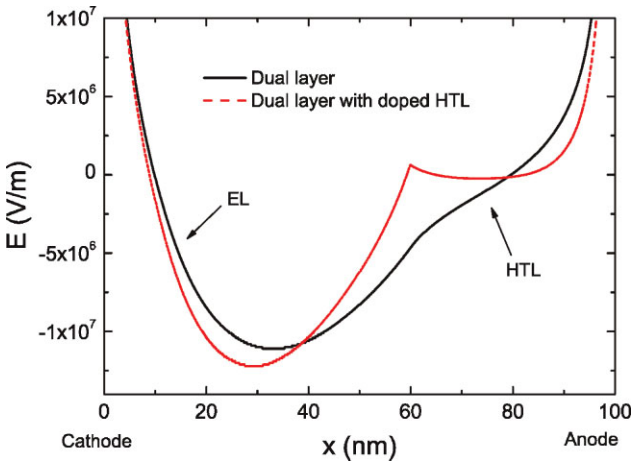

Figure 6 (online colour at: www.pss-a.com) The calculated electric field distribution in the double layer devices with different p_0: 0 (solid), $2 \times 10^{23}\,m^{-3}$ (dash).

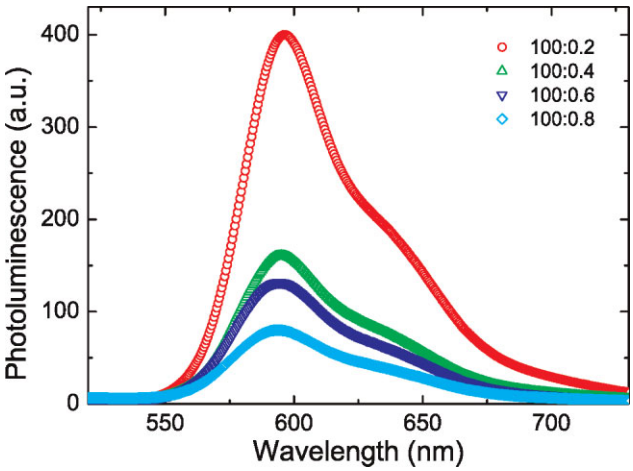

Figure 7 (online colour at: www.pss-a.com) Photoluminescence spectra of F4TCNQ doped MEH-PPV thin films according to different doping ratio. All the thin films have the same thickness: 140 nm.

is a voltage drop across this layer. For p_0 is $2 \times 10^{23}\,m^{-3}$ the electric field across the HTL is close to zero, showing that the voltage drop over this layer is small. This confirms that the doping process improves the hole conduction of the HTL. However, it is also observed that near the contacts the electric field changes sign due to the diffusion of charge carriers into the HTL from the Ohmic contacts [29]. Already at zero bias, charges diffuse into the polymer layer within 10–20 nm from the contacts. When the layer thickness is small, this background density of diffused charges can have a pronounced effect on the average charge density across the layer. It has been shown that, for a thickness of ~40 nm the average charge carrier density at zero bias is already in the order of $10^{22}\,m^{-3}$ [32]. The HTL can therefore be considered to be 'doped' by the charges diffused into the layer from the contacts. As a result, the difference in device current between the doped and undoped double layer devices is strongly reduced.

The decrease in efficiency for the double layer devices requires a different explanation. As for the undoped double layer device, a possible explanation for the decreased maximum efficiency is the overlap in the absorption and emission spectra (Fig. 3), which is small, however. Since MEH-PPV has the same HOMO and LUMO levels as BEH/BB-PPV (1:3), there is no energy barrier expected between the EML and the HTL [16]. When the applied bias is high, electrons will be injected into the HTL and recombine there with holes. Since the PL efficiency of BEH/BB-PPV is lower as compared to MEH-PPV, inefficient recombination in the HTL cannot be excluded. This can also explain the efficiency decrease for the double layer device upon doping the HTL when excitons formed in the HTL are quenched by the dopant. To study the quenching by the dopant, MEH-PPV solutions with different doping concentrations are spin casted on glass substrates. The photoluminescence (PL) spectra (Fig. 7) show that the PL intensity decreases with increasing doping concentration, confirming the quenching effect of the dopant. In an ideal multilayer PLED, the HTL (or ETL) would be designed as the electron (or hole) blocking layer. In this case all the charge carriers will be confined inside the EML and cannot enter into the transport layers.

A closer look into the efficiency–voltage characteristics reveals other features: The efficiencies of the 60 nm single layer device and the double layer device with doped HTL both reach their maximum efficiency at the same voltage (~2.5 V). This indicates that for both devices most of the voltage drop is across the MEH-PPV layers, which have the same thickness. As a result, at low bias voltages the current, luminance and (power) efficiency for the double layer device with doped HTL are higher than the device without doping. At higher applied bias, the current of the two double layer devices start to approach each other. Under these operating conditions, the amount of charge carriers that is induced by doping in the HTL will be outnumbered by the amount of charge carriers that are injected from the contacts. The efficiency of the device with the doped PLED becomes lower due to the additional quenching of the luminescence by the dopant. This can be overcome by using HTL layers that also have an electron blocking functionality, which is a subject of further study.

4 Conclusion Solution processed double layer PLEDs were fabricated using an orthogonal solvent system: the first polymer layer was spin casted from a chloroform solution, the second layer from a toluene solution. The conductivity of a BEH/BB-PPV (1:3) HTL was observed to increase upon p-type doping with F4TCNQ. Smooth and uniform p-doped polymer layers were achieved by using NMP as the solvent for the dopant, preventing aggregation in the polymer/dopant solution. The doped HTL was observed to lower the operating voltage of a double layer PLED as compared to its undoped counterpart. By device modelling an accurate description of the *J*–*L*–*V* characteristics could be obtained, explaining the observed results.

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